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A PRELIMINARY EVALUATION OF TACOT,
A NEW HEAT RESISTANT EXPLOSIVE (U)

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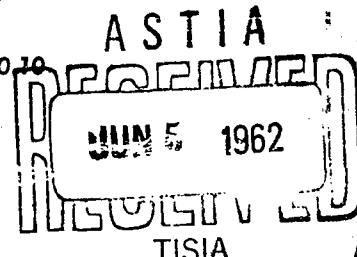
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A PRELIMINARY EVALUATION OF TACOT, A NEW HEAT RESISTANT EXPLOSIVE (U)

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ABSTRACT: TACOT and TATB have the lowest thermal decomposition rates of a series of heat-resistant explosives compared by means of 2-hour exposures to 260 and 280°C. The order of resistance of small quantities to rapid, complete decomposition on sudden exposure to temperatures above 300°C is TATB, TACOT, DATB. TACOT is more easily initiated by explosive shock than DATB and is considerably easier to initiate than TATB. TACOT and DATB have similar detonation properties; preliminary data indicate that the plate push velocity of TACOT is 5 to 10 percent lower than that of DATB. Solvent pressing considerably improves the density of TACOT charges and pre-ejection cooling reduces mechanical failures. The maximum attainable pressed density for the present form of TACOT appears to be about 95% TMD.

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The high temperature environment of guided missiles, particularly that created by long-time aerodynamic heating due to carry by supersonic aircraft, has stimulated a demand for high explosives which have superior ability to withstand such an environment. The Naval Ordnance Laboratory has synthesized a number of explosive compounds in its Foundational Research program which have the desired characteristics. One, diaminotrinitrobenzene (DATB), has been released for use in the SPARROW air-to-air missile.

This report evaluates a new candidate heat resistant explosive, TACOT, developed by the du Pont Company. Comparisons are made with other common and experimental explosives on the basis of small scale laboratory tests which have been used by this Laboratory for some years. Every effort was made to assure that measurements were made under comparable conditions. Due consideration must be given to the small scale and small number of determinations, as well as technical difficulties which are discussed in the report such as (a) the poor consolidation characteristics of TACOT and (b) the problem of obtaining high purity materials. The conclusions are considered valid within these limitations.

The work was done under the Explosives Research Task No. RUME-3E-000/212-1/F008-10-004, problem assignment No. 012. It will be useful to researchers within the military establishment who are interested in the properties of heat resistant explosives and to development engineers charged with the responsibility of selecting explosives for missile applications.

Users of the report are cautioned to protect any proprietary rights the E.I. du Pont Company may have in the compound, TACOT.

Dr. Donna Price, Physical Chemistry Division, and Mr. B. Drimmer, Explosion Dynamics Division, reviewed the report and made a number of helpful suggestions. Dr. J.M. Rosen, Organic Chemistry Division, and Mr. J.N. Ayres, Explosion Dynamics Division, contributed advice in the interpretation of some of the data.

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Mr. Harry Heller, Chemical Engineering Division, conducted the pressing studies. Mrs. Sarah Duck, Chemical Engineering Division, measured the impact sensitivities and Mr. H. Simmons, Organic Chemistry Division, measured the vacuum stabilities. Mr. J. A. Roslund, Explosion Dynamics Division determined the plate push velocity of TACOT.

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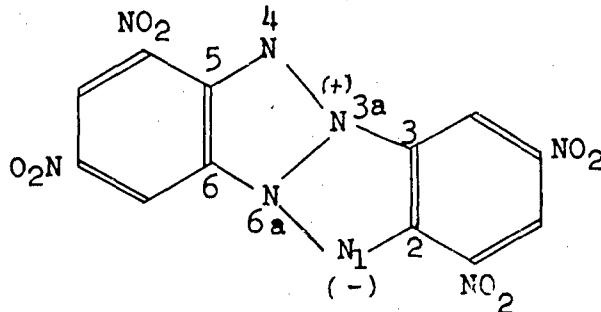
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A PRELIMINARY EVALUATION OF TACOT, A NEW HEAT RESISTANT EXPLOSIVE (U)

1.0 Introduction

1.1 The results of an initial evaluation of a new heat resistant compound, trade named TACOT, are assembled in this report. According to the supplier, the E.I. du Pont Company, the compound has the following structure:



$C_{12}H_4N_8O_8$, Molecular Weight = 388

The compound has been tentatively named tetranitrodibenzo-1,3a,4,6a-tetrazapentaline by the supplier. Information about TACOT received from the du Pont company is given below.

Crystal Density	1.84 to 1.86 gm/cm ³
Single Crystal Decomposition Temperature	410°C
Heat of Combustion	3575 cal/gm
Detonation Velocity	
In Aluminum Sheaths	6600 m/sec
In Pellet Form	7300 m/sec

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TACOT is purified by crystallization from 95% nitric acid in which its solubility is approximately 59% by weight.

1.2 The Naval Ordnance Laboratory's evaluation covered thermal stability, impact sensitivity, booster or card gap sensitivity, and detonation properties. Available data are presented for comparison of TACOT with TNT, DATB (1,3-diamino-2,4,6-trinitrobenzene), TATB (1,3,5-triamino-2,4,6-trinitrobenzene), TNB (sym-trinitrobenzene) and HMX. The detonation properties of PBX 9404, a plastic-bonded HMX composition, are also included in Table 4. A comparison between TATB and TACOT is of particular interest since both retain appreciable thermal stability at temperatures above the rapid decomposition area for DATB, approximately 280°C, for exposures over 60 minutes. A report in preparation by the Explosion Dynamics Division will give a more detailed interpretation of TACOT booster sensitivity relative to that of other heat resistant explosives of current interest, including DATB and TATB. Appendix A gives a list of references describing procedural details of tests used to obtain data quoted and discussed in this report.

2.0 Thermal Stability

2.1 In vacuum thermal stability testing small samples are heated at a fixed temperature below the point of rapid, complete decomposition. Decomposition rates which are measured are strongly influenced by purity of the sample so that thermal stability performances of the heat resistant explosives being considered here must be interpreted in this light. There is some variation in stability data for DATB attributable to differences in purity of the samples tested. Table 1 presents thermal stability data for the more stable samples of DATB. A solid-solid phase change occurs in DATB at 217°C(6), though none has been found in TATB. An inflection in the derivative differential thermal analysis curve for TACOT suggests the possibility of a solid-solid phase transition but this has not been confirmed by other tests so far.

2.2 Recent analyses of pilot plant production TATB for thermal stability showed a gas evolution of 2 cc/gm/hr for a 2-hour period at 280°C. TATB from this same batch began autocatalytic decomposition at 300°C in 95 minutes; TACOT began autocatalysis at 300°C in 150 minutes. The thermal stability of TATB is known to be sensitive to the presence of impurities but the exact nature of the impurities involved is not completely understood. The most efficient procedure for preparing TATB at present is based on successive nitration and amination of 1,3,5-trichlorobenzene and there is a possibility of by-product influence. Available data indicate that TACOT can resist the

development of autogenetic decomposition at 300°C for a somewhat longer period of time than TATB in its present state of development. At 280°C both TACOT and TATB exhibit a high order of heat resistance and can be considered stable during the 2-hour duration of the test. It is not the intent here, however, to make a final resolution of differences between the two compounds with respect to thermal stability because of limited data.

2.3 Amine-free symmetrical trinitrobenzene (TNB) may possess the highest degree of thermal stability obtainable from the resonance-stabilized nitrated benzene nucleus. At 260°C, TNB has the stability of TACOT and a recent differential thermal analysis of TNB at this Laboratory showed boiling without measurable decomposition, within the limits of accuracy of the instrumentation, at approximately 340°C.

3.0 Melting Points and Ignition Temperatures

3.1 The term ignition as used here refers to rapid, complete decomposition with or without appearance of flame. Ignition data obtained by different methods are not comparable and therefore have not been tabulated. Available ignition data are mentioned here, however, as an index of temperatures and conditions that will produce rapid, complete decomposition in the heat resistant explosives being compared.

3.2 TNT, DATB and TNB melt appreciably below their deflagration temperatures. With rising temperature, TATB, TACOT and HMX decompose either without melting or with the formation of short-lived liquid decomposition products, Table 1. A crystal of TATB decomposes near 450°C when heated 2 to 3°C per minute from sudden exposure near 440°C (7). The single crystal decomposition point for TACOT is 410°C, determined by slow heating from ambient temperature (1). Deflagration temperatures of 314°C for DATB and 225°C for TNT have been determined with 2-pound slabs heated at 100°C/min on one surface (5).

3.3 On sudden exposure, TACOT ignites in 15 minutes at 337°C and in 0.1 sec between 388 to 400°C (1). Time to ignition after sudden exposure is plotted as a function of temperature in Figure 1 for DATB and TATB. The curve for DATB is based on the disappearance of the sample by sublimation rather than by ignition. Small quantities of DATB sublime rapidly without apparent decomposition when suddenly exposed to temperatures well above the point of deflagration of a 2 pound slab. On exposure to 335°C, a 200 mg sample of DATB rapidly disappears, either subliming

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(or boiling off) completely in 5-seconds without apparent decomposition. The 5-second decomposition temperature for 100 mg of TATB is 510°C as shown by the appearance of yellow vapor. The order of resistance to rapid, complete decomposition on sudden exposure to high temperature appears to be TATB, TACOT, DATE on the basis of available data. This represents order of stability at very high heating rates for short periods. At lower heating rates, order of heat resistance is better described by performance in the vacuum stability test.

4.0 Sensitivity to Initiation by Explosive Shock

4.1 The relative sensitivity of TACOT to high shock loads was determined by a gap test using a test column 0.2-inch diameter and 1.5-inch long contained in a 2 inch diameter brass mold. A standard RDX booster separated from the test charge by varying lengths of Lucite was used to determine the gap required for initiation of 50% of the charges. The 50% fire point is given in Table 2A in both decibang (DBg)** units and in actual Lucite-gap thicknesses, longer gaps corresponding to greater sensitivity.*** The Lucite gap for RDX, 390 mils at 93% TMD*, indicates that RDX initiates high order in 50% of the charges when booster and RDX are separated by 390 mils of Lucite. Since shock attenuation increases approximately exponentially with gap distance, logarithmic gap distances expressed in DBg values are believed to be a better measure of the minimum boostering requirement.

4.2 Table 2A compares the shock sensitivity of TACOT and 5 other explosives at 93 and 95% TMD. When vacuum pressed into the test fixture at 64,000 psi TACOT has a density of 93% TMD (1.71 gm/cm³) and sensitivity data for the other compositions were interpolated at this point for comparison. At 93% TMD, TACOT is not as sensitive to initiation by explosive shock as TNT but is more sensitive than DATB and appreciably more sensitive than TATB. TACOT sensitivity data were not extrapolated to 95% TMD because the slope of the density-sensitivity curve may increase sharply at densities approaching theoretical. Data on other compositions in Table 2A were either interpolated or determined at 95% TMD. As shown in Table 2A, the decrease in sensitivity with rising density is greatest in TATE. Though the sensitivity-density curve for TACOT (Figure 2) cannot be extrapolated it is probable that TACOT does not change its

* Theoretical Maximum Density

** A Decibang Unit is one Unit of the Function "30-10 log (test gap/ref gap)" (4).

*** See Figure 3

relative position in Table 2A between 93 and 95% TMD. Recent pressing experiments (Sec. 7.0) produced pellets of pure TACOT near 95% TMD so that the sensitivity of TACOT at this density is of interest. Table 5, which lists densities attainable at the indicated loading pressures, shows that, except for TACOT, the tabulated compositions are pressable to densities above 95% TMD.

4.3 Gap sensitivity as a function of density is shown in Table 3 and Figure 2 for TACOT, DATB and DATB/Zytel 95/5. The greater sensitivity of TACOT is apparent in Figure 2 up to the maximum TACOT density reached. The slope of the plot suggests that at higher densities TACOT booster sensitivity may approach those of DATB and DATB/Zytel 95/5. As expected DATB is more sensitive than DATB/Zytel 95/5 at lower densities but above 93% TMD, the difference becomes extremely small (2). Unfortunately gap sensitivity data on TACOT/Zytel 95/5 is not available for this evaluation though the detonation properties of this composition were measured and are discussed in Sec. 6.0. A continuation of the TACOT study, if undertaken, should include a gap sensitivity-density plot for TACOT/Zytel 95/5.

5.0 Impact Hammer Sensitivity

5.1 Trinitrobenzene and TACOT have 50% initiation heights very near 100 cm, while DATB and TATB show the stabilizing effect of amino substitution on the nitrated benzene nucleus with respect to impact sensitivity, Table 2B. Though not shown in Table 2B, trinitroaniline (Picramide) has a 50% impact height of 177 cm, between that of TNB and DATB; the 50% impact height therefore increases regularly in this series with increasing amino substitution.

6.0 Detonation Properties

6.1 Detonation velocities and energies given in Table 4 are based on rate stick measurements; detonation pressures are derived from water shock experiments. Charges of TACOT on which detonation property measurements were made had densities below 90% TMD because they were prepared prior to the solvent pressing study (Sec. 7.0). A TACOT rate stick having an average density of 1.45 gm/cm³ (78.8% TMD) gave a photographically-measured detonation velocity of 6448 m/sec. A TACOT wedge, maximum thickness 12.7 mm, density 1.59 gm/cm³ (86.5% TMD), built up to a detonation velocity of 6690 m/sec in the NOL wedge test, Appendix A (3c). On the basis of available data the best evaluation of TACOT performance can be made by comparing TACOT/Zytel, 95/5

(95.5% TMD) with DATB/Zytel, 95/5 (96.5% TMD) in Table 4 (3). The performances of these two plastic-bonded explosives are very similar: detonation velocities, 7128 m/sec for TACOT/Zytel vs 7200 m/sec for DATB/Zytel; detonation pressures: TACOT, 209 kbar vs 224 kbar for the DATB mixture. The detonation energy for TACOT, 742 cal/gm, was some 7% lower than the 792 cal/g found for the DATB mixture.

6.2 The plate push performances of all compositions in Table 4 were calculated for 93% TMD from experimental data at other densities (except where indicated that the measurement was made at this density). Because TACOT plate push performance was measured at an unusually low density (86.5% TMD) the accuracy of the value calculated for 93% TMD may be below that of other plate push data in Table 4. However, the lower detonation energy of TACOT as compared to DATB, would imply that the 2725 ft/sec value calculated for TACOT (93% TMD) would probably be a good estimate, since plate-push velocities correlate reasonably well with detonation energies.

7.0 Pressing of TACOT and TACOT/Zytel 95/5

7.1 The best 0.5 and 1.0-inch diameter pellets of pure TACOT which were vacuum pressed dry at 20,000 psi and at various temperatures, 50 to 150°C, had densities near 86% TMD and tended to crack on ejection. Ejection at elevated temperatures usually produced defective pellets. A limited number of experiments described in Table 6 suggest it may be possible to press TACOT to 95% TMD and obtain pellets resistant to mechanical breakdown by wetting the powder with acetone and cooling the pressed pellets to room temperature before ejection. Another method has been suggested for pressing TACOT near 95% TMD without mechanical failure of the charge: slow application of high pressure, long dwell, 48 hours or more, and heat. This technique has been reported by the E.I. du Pont Company Explosives Division Laboratories (1).

7.2 Some pellets of the composition TACOT/Zytel 95/5 could be pressed to densities near 95% TMD. Zytel is a grade of Nylon used in explosive molding powders as a bonding agent. Table 7 compares laboratory pellets pressed at 30,000 psi at various temperatures, with and without pre-ejection cooling. Though it is apparent that a reasonably high density pellet with good mechanical properties can be prepared without pre-cooling, further work may establish that pre-cooling improves density uniformity.

8.0 Conclusions

8.1 Of the compositions shown in Table 1, TACOT and TATB have the lowest decomposition rates in the vacuum thermal stability test at 260 and 280°C, based on 2-hour exposures.

8.2 The order of resistance of small quantities to rapid, complete decomposition on sudden exposure to temperatures over 300°C is TATB, TACOT, DATB. This represents order of stability at very high heating rates for short periods.

8.3 In the small scale gap test, RDX and TNT are more shock sensitive than TACOT. TACOT is more sensitive than DATB and considerably more sensitive than TATB. Above 90% TMD, TACOT and DATB are about equivalent in rate of sensitivity change with density.

8.4 On the basis of performance in compositions with 5% Zytel binder, TACOT and DATB have similar detonation velocities and pressures. Preliminary plate-push data indicate that the plate-push value for TACOT is between 5 and 10 per cent lower than the value for DATB.

8.5 The density of small charges of pure TACOT can be considerably improved by solvent pressing. Mechanical failure of charges can be reduced by pre-ejection cooling to ambient temperature in the mold. The maximum attainable density with available material and presses appears to be about 95%. TACOT and Zytel blend readily to form a plastic-bonded explosive pressable to 95% TMD.

9.0 Recommendations

The following additional work is recommended to fill gaps in the available information shown in Table 4.

- (a) Obtain vacuum thermal stability data above 300°C for TACOT.
- (b) Determine small scale gap sensitivity of TACOT/Zytel 95/5.
- (c) Measure the plate push velocity at 95% TMD for TACOT, TACOT/Zytel 95/5 and DATB/Zytel 95/5.
- (d) Run the wedge test on pure TACOT at 95% TMD, providing sufficiently large pellets can be pressed to this density. This information will make possible a comparison of the build-up-to-detonation of TACOT and DATB.

TABLE I

Melting Point (°C)	STABILITY				HMX 285(2)
	TNT	TNB	DATB	TATB	
81	121	286(7)	450(2)	410(2)	
Vacuum Stability 100°C (cc/gm/48 hrs) 260°C (cc/gm/hr) 280°C (cc/gm/hr)	<0.1 - -	<0.10 - -	0.0 3 to 4 52.5	0.0 0.7 2.0	0.0 0.1 0.3
Heat of Formation (4) (kcal/mole)	-17.81	-11.40(5)	-29.23	-36.85(6)	- - +17.93

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- (1) Average for 2-hr exposure
- (2) Decomposes
- (3) Average for 5-hr period
- (4) (-) exothermic
- (5) Reference (8)
- (6) Reference (9)
- (7) Reference (6)

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TABLE 2A
SMALL SCALE GAP (BOOSTER) SENSITIVITY TEST

Pressed Compositions	DB _g (2)	At 93% TMD(1)		DB _g (2)	At 95% TMD(1)	
		Lucite Gap (mils)	Lucite Gap (mils)		Lucite Gap (mils)	RDX
RDX	4.1	390	5.0	316		
TNB	5.2	303	5.7	269		
TNT	5.7	269	6.0	251		
TACOT	7.5	182	--	--		
DATA	8.4	145	8.8	132		
TATB	10.1	98	11.1	78		

(1) TMD = Theoretical Maximum Density

(2) DB_g = 30-10 log (test gap/ref. gap)

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TABLE 2B
IMPACT HAMMER SENSITIVITY

50% Initiation ht. (in. cm.)	TNT	TNB	DATA	TATB	TACOT	RDX
150 to 215	100	>320	>320	101	16 to 33	

TABLE 3

SENSITIVITY CHANGE WITH DENSITY

Loading Pressure PSI	Density (gm/cm ³)	%TMD(1)	TACOT	DB _g (2)	Lucite Gap in Millis
4000	1.1620	63.2		5.56	278
8000	1.2809	69.1		5.64	273
16000	1.4345	78.0		6.06	248
32000	1.5928	86.6		6.70	214
64000	1.6983	92.3		7.46	180
			<u>DATA</u>		
				6.938	202
				7.375	183
				7.875	163
				8.100	155
				9.000	126
			<u>DATA/Zytek 95/5</u>		
				7.45	180
				7.74	168
				8.03	157
				8.46	143
				9.33	117

(1) TMD = Theoretical Maximum Density
 (2) DB_g = 30-10 log (test gap/ref. gap)

TABLE 4

DETONATION PROPERTIES

TNT (cast)	TNB	DATB	TATB	TACOT	PBX 9404(7)	TACOT 95 Zytel 5	DATB 95 Zytel 5
TMD(2)	1.651	1.688	1.837	1.938	{ 1.84- 1.86 }	1.903	1.75
Experimental Density (gm/cm ³)	1.619	1.640	1.800	1.802	{ 1.45 }	1.850	1.67
%TMD(2)	98.1	97.2	98.2	95.0	78.8	97.0	95.5
Detonation Properties (4)							96.5
Velocity (m/sec)	6780	7269	7600	7658	6448	8724	7128
(dD/dt) ² (m/sec/gm/cm ³)	3225	2852	2852	2852	--	--	--
Pressure (kbars) (1)	192	214	251	259	--	333	209
Energy (cal/gm) (3)	780	766	800	829	--	1022	742
Isentropic Exponent, k	2.81	3.04	3.10	3.07	--	3.15	3.17
Plate Push Value (ft/sec)							
At 98% TMD(5)	2930	2946	3130	3229	--	3730{6}	--
At 93% TMD(5)	2830(9)	2841	3028	3124	2725(8)	3680{6}	--
Failure Diameter (cm)	2.69(10)	<0.3	0.53	1.3	<0.3	0.1 to 0.2	--

(1) Water Shock Measurement
(2) Theoretical Maximum Density
D²/2(K²-1)

(3) At Experimental Density

(4) Calculated from Values Measured at other Densities
(5) Measured at the Indicated Density
HMX 94%, Pyrocellulose 4%, Trichloroethylphosphate 2%
(6) Calculated from a Measurement at 86.5% TMD
(7) Measured with Cast TNT Powder Pressed to 93% TMD
(8) Failure Diameter of Pressed TNT at 98% TMD is 1/10 of this Value(10)

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TABLE 5

PRESSED DENSITIES AT INDICATED LOADING PRESSURES

<u>Composition</u>	<u>Pressed Density (gm/cm³)</u>	<u>%TMD(1)</u>	<u>Loading Pressure (PSI)</u>
RDX	1.73	96.0	38,200
TNT	1.62	97.5	10,000(75°C)
TACOT	1.71	93.0	64,000
TNB	1.69	100.0	64,000
DATB	1.76	96.0	64,000
TATB	1.89	97.6	64,000

(1) TMD = Theoretical Maximum Density

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TABLE 6

VACUUM PRESSING OF PURE TACOT PELLETS 1/2" DIAMETER x 1/2" LONG, AT 20,000 PSI

Temp (°C)	Dwell (Min)	Ejection Temp (°C)	Results (gm/cm ³)	Density	%TMD	Solvent
150	20	50	Cracked	- -	- -	None
140	20	50	Good	1.56	85.0	None
150	20	25	Cracked	1.56	85.0	None
13.	30	25	Good	1.72	93.5	3% Methylene Chloride
50	30	25	Good	1.75	95.0	3% Acetone
60	30	25	Cracked	- -	- -	3% Acetone
60	45	25	Good	1.76	95.5	3% Acetone

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TABLE 7
VACUUM PRESSING OF TACOT/Zytec 95/5 AT 30,000 PSI AND 120°C
DWELL TIME 10 MINUTES

Ejection Temperature (°C)	Pellet Size Diam. & Length (in)	Results	Density (gm/cc)	%TMD
120	1/2 x 1/2	Good	1.64	92.1
95	1/2 x 1	Good	1.69	94.9
95	1/2 x 1/2	Good	1.65	92.6
25	1 x 1	Good	1.67	94.0
25	1/2 x 1	Good	1.71	95.5
120	1 x 1	Good	1.65	93.0
95	1 x 1	Good	1.65	93.0

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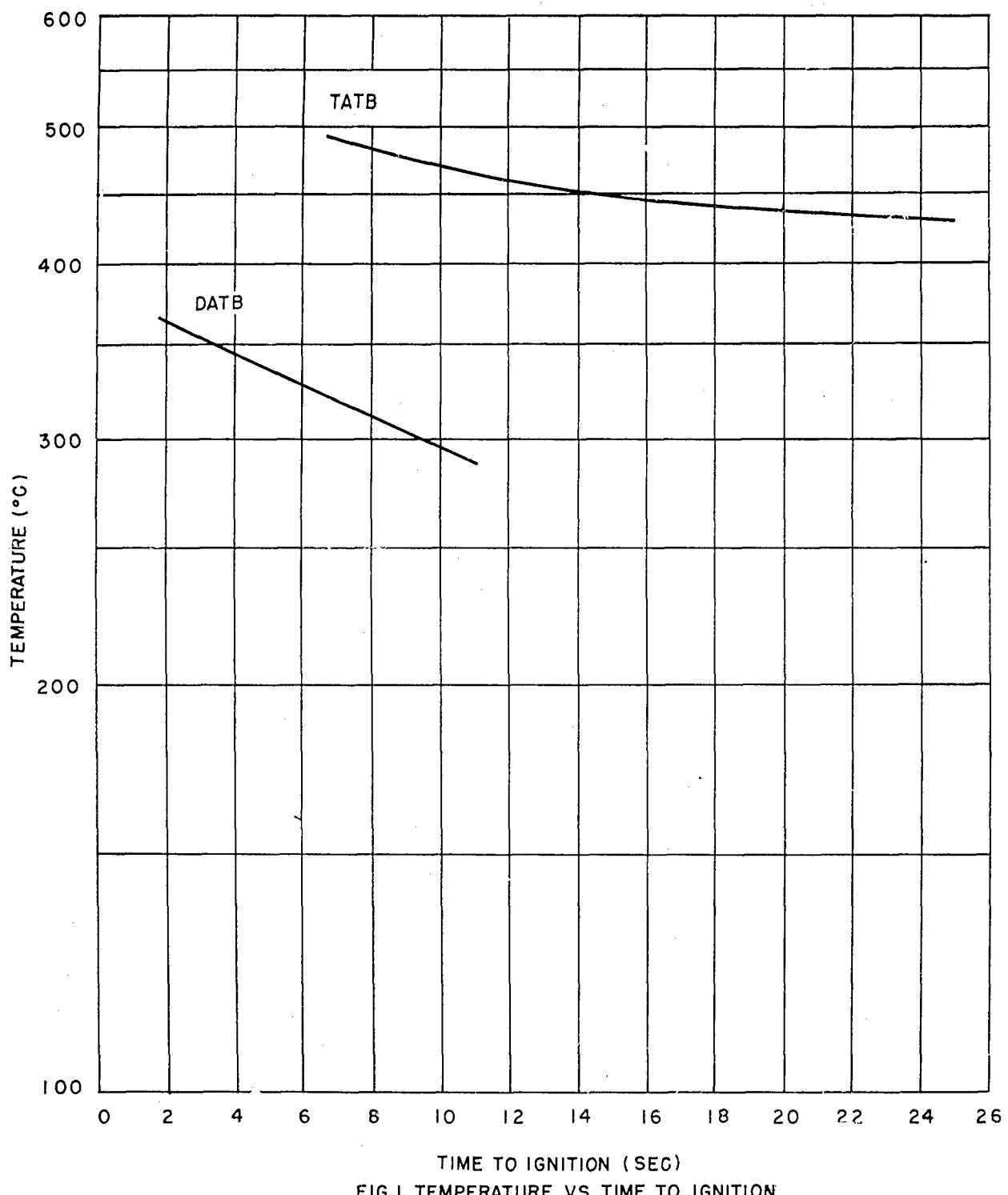


FIG.1 TEMPERATURE VS TIME TO IGNITION
FOR DATB AND TATB

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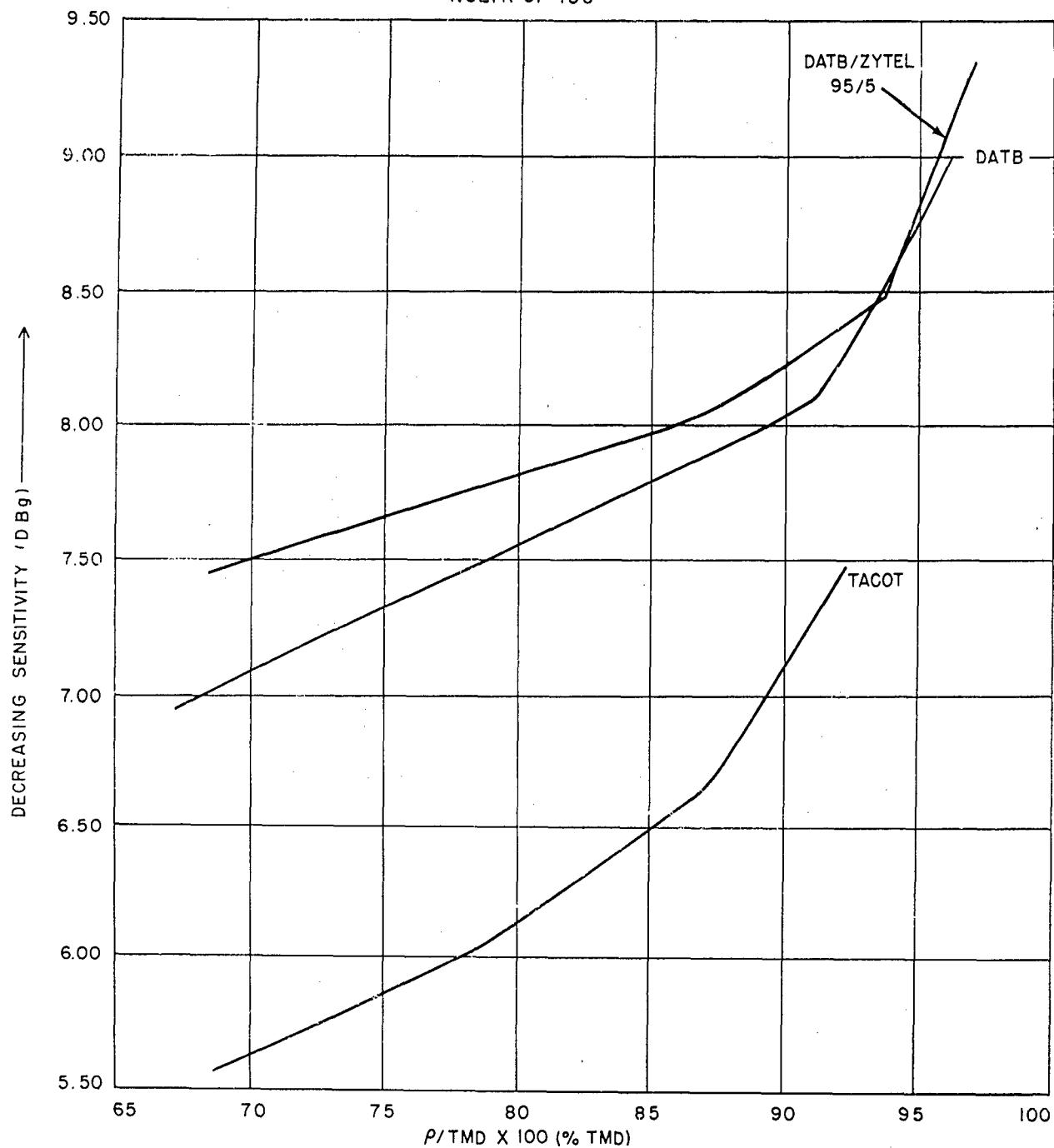


FIG. 2 EFFECT OF DENSITY ON THE GAP SENSITIVITY
OF TACOT, DATB AND DATB/ZYTEL 95/5

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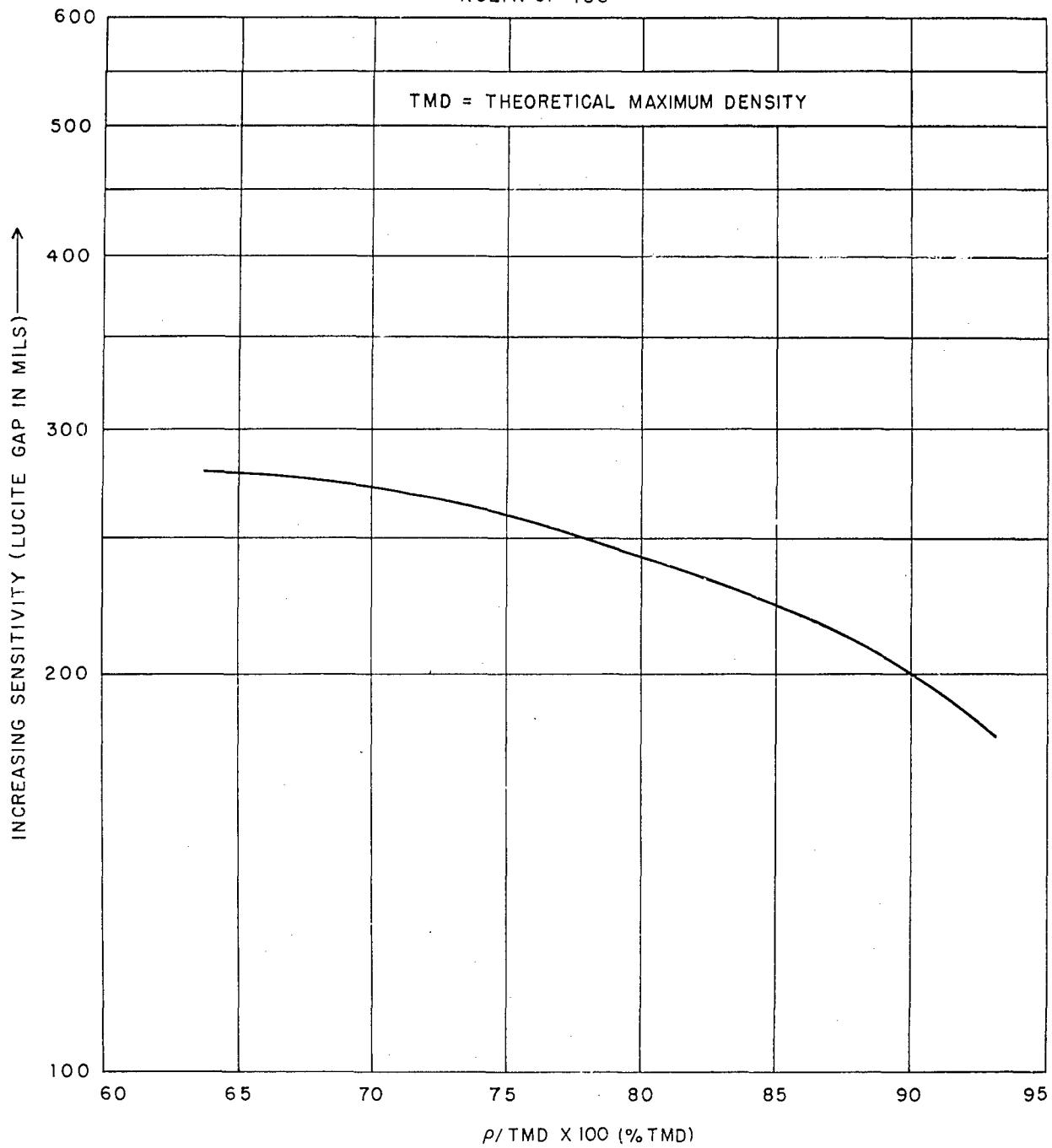


FIG. 3 EFFECT OF DENSITY ON THE GAP SENSITIVITY
OF TACOT EXPRESSED IN GAP DISTANCE

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REFERENCES DESCRIBING TEST PROCEDURE AND APPARATUS

Appendix A

References describing procedures and in some instances the apparatus for obtaining sensitivity, stability, detonation property and pressing data presented in this report are given here.

1. Vacuum Thermal Stability

NAVORD Report 6629, "Improved Apparatus and Technique for the measurement of the Vacuum Stability of Explosives at Elevated Temperatures", A.H. Rosen, H. Simmons, 12 March 1959

2. Sensitivity

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3. Detonation Properties

(a) Detonation Pressure

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(b) Detonation Velocity

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4. Pellet Pressing

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